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Quantification and comparison of Halo-acetic acids formation potential in Ganga River Water Samples with diluted waste water samples using Gas Chromatography

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ABSTRACT

Treated drinking water supply systems in the world rely on natural water bodies like rivers and ground water which are continuous sources for raw water. The natural water from these sourcescontains a complex mixture of natural organic matter (NOM), pathogens, different types of ions etc., which when react with chlorine while treatment for potable drinking water has the potential to generate carcinogenic disinfection by-products (DBPs) such as Halo-acetic acids (HAAs). The main objective of this study is to investigate the formation of the nine major Halo-acetic acids (HAAs) viz. monochloroacetic acid (MCAA), dichloroacetic acid (DCAA), trichloroacetic acid (TCAA), monobromoaceticacid (MBAA), dibromoacetic acid (DBAA), tribromoacetic acid (TBAA), chlorobromo acetic acid (CBAA), dichlorobromoacetic acid (DCBAA), dibromochloroacetic acid (DBCAA),in Ganga river water. In this present study; formation potentials of nine HAAs in Ganga water was investigated and compared the same with diluted waste water samples.Liquid-liquid extraction method, followed by qualitative and quantitative estimation by gas chromatograph (BUCK GC 990) equipped with electron capture detector was used for the experiment.
From the results obtained and the results observed in the literature it could be concluded that the organic matter present in the Ganga River water is mostly of anthropogenic i.e., effluent origin organic matter (EffOM);which directly indicating the possibilities for presence of large quantities of carcinogenic disinfection by-products (DBPs) in supplies from drinking water treatment plant at Kanpur, UP, India and also traditional water treatment techniques like alum coagulation and chlorination is no more a viable solution for safe health.

1) INTRODUCTION

Chlorinated disinfection by products (DBPs) are formed when chlorine is added as a disinfectant to water containing organic matter either from natural or anthropogenic sources[1,2,3]. Trihalomethanes (THMs) and haloacetic acids (HAAs) are the two major classes ofcommon chlorinated DBPs in precise $CHCl_3$ and $CHCl_2Br$ are the most common THMs, and DCAA and TCAA are the most common HAAs reported in chlorinated water supplies. Furthermore, for a constant bromide concentration in raw water, increases in the chlorine dose shift the formation of DBPs to the less bromine-substituted species [4]. The effect of bromide ion on the formation and speciation of trihalomethanes (THMs) and haloacetic acids (HAAs) during the chlorination of biologically treated wastewaters shows that the formation of total THMs and total HAAs during chlorine disinfection increases with increasing bromide levels in wastewater [5].
Several studies and reports have indicated that elevated concentrations of chlorinated DBPs, especially that of THMs and HAAs, cause cancer in animals [1,6]. The United States Environmental Protection Agency (USEPA) has limited the maximum contaminant level (MCL) of total trihalomethanes (TTHMs) in municipal water supplies to 0.08 mg/L and total of five HAAs (CAA, DCAA, TCAA, BAA, and DBAA) to 0.06 mg/L. THMs are characterized by USEPA as Group B carcinogens, i.e., known to cause cancer in laboratory animals. Chloroform is by far the most common THM observed in chlorinated water while brominated THMs being only prevalent in waters containing high concentrations of bromide. $CHClBr_2$ constitutes the most serious cancer risk, followed by $CHBr_3$, and $CHCl_3$. HAAs are also classified by the EPA as Group B carcinogens [6].
The work carried on assessment and control of chlorinated DBPs during water or wastewater disinfection in India is meagre and not sufficient to set standards and policies for safe health in India. Thus there is a need for sensitizing regulatory authorities and public in general regarding long-term harmful

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impacts of drinking water with high chlorinated DBPs concentrations. A study conducted in Chennai showed that the total trihalomethanes level (TTHM) exceeds the EPA's maximum contaminant level in many cases [7]. In a study conducted in Mumbai the levels of $CHCl_3$ found in the post-monsoon season were,Ghatkopar (226 µg/L), Malabar (210.3 µg/L) and Tulsi (231.26 µg/L), which are all above EPA MCL for TTHMs [8]. Considering that the surface water sources supplying the raw water to water treatment plants in India are often polluted with high concentrations of organic matter from anthropogenic sources, it is expected that chlorination of treated water in most Indian water treatment plants will result in the formation of high concentration of DBPs.

2) OBJECTIVES

The broad objective of the present study was to investigate the extent of DBPs formation on the chlorination of water from Ganga River at Kanpur and draw conclusions regarding the nature of DBP precursors and also the feasibility of DBP precursor removal from this water by the coagulation process during water treatment.

Specifically, the main tasks performed during the study were the following:

1. Determination of the extent of HAAs formation when Ganga river water (GW) collected from the intake to the Ganga barrage water treatment plant in Kanpur is chlorinated at various chlorine doses after primary sedimentation.
2. Determination of extent of HAAs formation when domestic wastewater collected from the intake of the Jajmau wastewater treatment plant in Kanpur was diluted and chlorinated at various chlorine doses after primary sedimentation.
3. Comparison the HAAs formation in the above two cases with respect to both the extent for HAAs formation and species distribution, followed by the drawing of appropriate conclusions regarding nature of organic matter present in GW.
4. Determination of the extent of HAAs formation when GW collected as above was settled and coagulated before chlorination at various chlorine doses, followed by drawing of appropriate conclusions regarding the efficiency of HAAs precursor removal from GW at Kanpur by the coagulation process.
5. Drawing appropriate conclusions regarding the methodology adopted for control of HAAs formation during chlorination of treated GW at Kanpur.

3) MATERIAL AND METHODS

Experimental Materials: Analytical reagent grade chemicals, High purity gas, De-ionized water obtained from a Milli-Q system (Millipore, USA) was used for all experimental purposes.

Chemicals: Pure compounds (>99%purity), viz., monochloroacetic acid, dichloroacetic acid, trichloro-acetic acid, monobromo acetic acid, dibromo acetic acid, tribromo acetic acid, chlorobromo acetic acid, dichlorobromo acetic acid, dibromochloro acetic acid, 1-2 dibromopropane, halo acetic acid mix (200 ppm) and Methyl-Tertiary-Butyl-Ether (HPLC grade, >99% purity) were purchased from Sigma–Aldrich Ltd., USA. Methanol (HPLC grade, >99% purity), Copper-II-Sulfate-V-hydrate (>99% purity) were purchased from E. Merck (India) Pvt. Ltd., Bombay. Sodium sulfite (>98% purity), anhydrous Sodium Sulfate (99.5% purity) and Sodium Bicarbonate (99.5% purity) were purchased from Loba chemicals, India. Alum i.e. aluminium sulphate, $Al_2(SO_4)_3.18H_2O$ (purity 99%), was purchased from Loba Chemicals, India.

Glassware: 40ml vials of Borosilicate glass (ASTM type-I, Wheaton Science, Millville, NJ, USA) equipped with screw caps having Teflon faced re-sealable septa were used. The 125ml capacity reagent bottles covered by aluminum foil (to serve as dark bottles) to preserve the chlorinated samples were also made of borosilicate glass. Micro syringes (Hamilton, USA) of 1µ L, 10µ L, 50µ L, 100µ L, 500µ L an d 1000µ L were used during the experiments. Clear 2ml gas chromatograph (GC) auto sampler vials with 11 mm aluminum seals and PTFE rubber lined septa (Wheaton Science, USA) were used for sample storage for Gas Chromatographic (GC) analysis. These vials were used only once before disposal. All other glassware used in this study were also made of borosilicate glass, and purchased from Borosil, India. Before use, glassware was acid washed, followed by rinsing with tap water and distilled water and then dried in an oven at 180˚C.

Glassware Washing: The washing protocol for the vials involved rinsing with tap water to remove sample or solvent residues as the first step. Then the vials were kept overnight in a chromic acid bath. Next, the vials were washed thoroughly with phosphate free laboratory detergent and rinsed thoroughly with tap water and later rinsed with deionised (Milli-Q) water. After washing, the glassware were kept in an oven at 180˚C for drying, and later stored in vacuum desiccators before final use. Re-useable septa of 40 ml vials were washed with phosphate free laboratory detergent and then thoroughly rinsed with tap water. They were rinsed with deionised water and kept in an oven at 50˚C for 2-3 hrs. Septa were stored in vacuum desiccators before use in experiments.

Sample Collection: Direct sampling method was used to collect water samples and standard operating procedure was followed in cleaning the sampling containers one day prior to go for sampling. Ganga River water was collected from the inlet of the water treatment plant at Ganga Barrage, Kanpur, by following direct sampling method. These samples were collected and stored at 4°C in the laboratory. A 1-liter water samples was allowed to settle in imhoff cone for 1 hour and supernatant used in various experiments.

Stock and Standard Solutions: 124.58g of Alum (aluminium sulphate) was diluted to1L with de-ionized water to prepare 10000 mg/L (as aluminium) alum stock solution.

Two stock solutions (each 200 mg/L) of glucose and sodium bicarbonate were prepared for total carbon (TC) and inorganic carbon(IC) measurement respectively. These stock solutions were diluted to prepare standards for the calibration of the TOC analyzer.

Pure compounds (the nine HAAs and the internal standard) were diluted to required concentrations using MTBE. Five point calibration curves were prepared for all HAAs. Sodium hypochlorite (1000 mg/L) stock solution was also prepared. The solution was diluted and used to add various chlorine doses to the Ganga water and wastewater samples to be chlorinated.

Coagulation and Flocculation: 1-liter aliquots of settled Ganga water or settled were taken in plastic jars. Alum was

added to the water from stock solution such that the alum dose was 60 mg/L (an optimum alum dose which was determined earlier according to standard procedure). Rapid mixing was done for one minute at 100 rpm, followed by slow mixing for 30 minutes. After mixing the jar was left undisturbed for 1 hour to allow settling of alum sludge produced. The supernatant, i.e., coagulated Ganga river water was used for various experimental purposes.

Chlorination of Samples: 150 mL of the water sample was taken in a BOD bottle and appropriate amount of chlorine was added such that the chlorine dose was either 4, 6 or 8 mg/L as Cl_2. After 30 minutes of chlorination, an aliquot of the chlorinated water was titrated by the DPD/FAS titration method to determine free and combined residual chlorine remaining in the sample. The remaining sample was stored at 4^oC and analyzed for determination of HAAs after 4 hours and 7 days of chlorination.

Extraction of HAAs: EPA Method 552.2 [9] was used for extraction of HAAs from the chlorinated water.

Gas Chromatography Analysis: Gas Chromatograph equipped with an electron capture detector (Model: 910, Buck Scientific, USA) was used to perform the chromatographic analyses. A capillary column 30 m x 0.53 mm Restek MXT^R-5 was used. Samples were extracted from GC autosampler vials using a micro syringe. Injection volume was 1 µL in all cases. High purity (zero grades) helium and nitrogen were used as the carrier and make up gases respectively. Carrier and make up gas flow were about 30mL/min and 60mL/min respectively. Chromatograms were analyzed using the 'Peak Sample 3.29' software. Injector, valve and the electron capture detector (ECD) temperatures were 150˚C, 150˚C and 200˚C respectively. The column oven temperature program for HAAs analyses were as shown in **Table 3.1**

Table 3.1GC Temperature Programs for HAAs Analysis

Analysis Type	Program	Program Duration
HAAs	Hold 1: 10 minutes at 40˚C Ramp 1: 40˚C – 65˚C at 6˚C / min Ramp 2: 65˚C –145˚C at 20˚C/ min Ramp 3: 145^{o}C–235^{o}C at 15^{o}C/min	24.2 minutes

4) RESULT AND DISCUSSION

Disinfection By-products (DBPs) Formation: Diluted wastewater (DW) samples with dissolved organic carbon values in the range of 1.5 – 15 mg L^{-1} were chlorinated with different chlorine doses, i.e., 3, 5, 8 and 12 mg L^{-1} (as Cl_2). Concentrations of various HAA components formed in each sample were measured after 7 days, by the time which DBPs formation reactions were assumed to be completed.

The extent of total HAA formation in samples containing no bromide are shown in Figure 4.1a, while the extent of total HAA formation in samples containing added bromide (~ 4.5 mg L-1) are shown in Figure 4.1b. Total HAA formation was more in samples containing bromide (Figure 4.1b) as compared to the corresponding samples containing no bromide (**Figure 4.1a**).

Based on the results presented in **Figure 4.1**, it was further apparent that presence of higher added chlorine concentration acted as a driving force leading to increased formation of HAAs. Thus for a particular DOC concentration, HAAs formation increased with increasing added chlorine dose. It was further noticed that HAAs formation levelled off at higher DOC concentrations, probably because the availability of chlorine became a limiting factor under these circumstances.

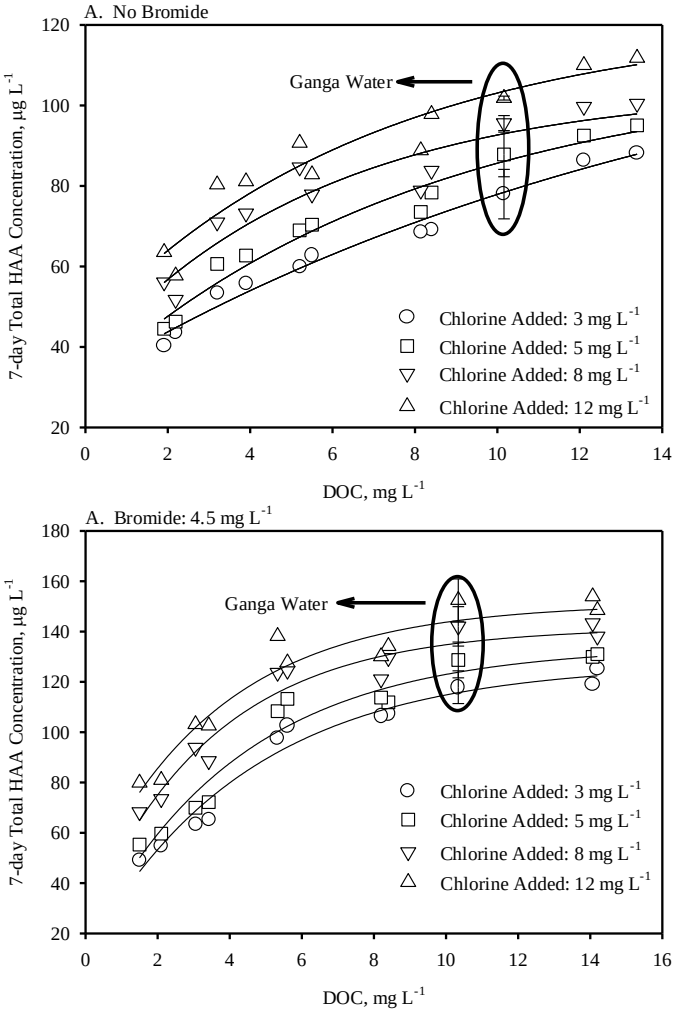

Fig 4.1 Total HAA Concentration measured after 7 days of Chlorination in Diluted Wastewater and Ganga River water at Various Applied Chlorine Doses. A) No Bromide; B) Bromide: 4.5 mg L^{-1}

Ganga water (GW) with DOC concentration of approximately 10 mg L^{-1} was chlorinated in a similar fashion as the DW samples. The total HAA formations after chlorination of GW samples were determined and are also shown in Figure 4.1 respectively for direct comparison with DW samples. Based on **Figure 4.1**, it may be concluded that at comparable DOC concentrations, the extent of HAAs formation in GW samples was similar to DW samples

Specific DBPs Formation: The 7-day HAAs formation value as reported earlier was normalized by dividing with the DOC of the sample to obtain the corresponding specific HAAs formation value.

The specific HAA formation values were also calculated for the DW samples and are presented in **Figure 4.2a** for the samples with no bromide and in **Figure 4.2b** for samples with added bromide. Based on the trends of the specific HAA

formation values presented in **Figures 4.2**, the specific HAA formation potential (HAAFP) can be calculated.

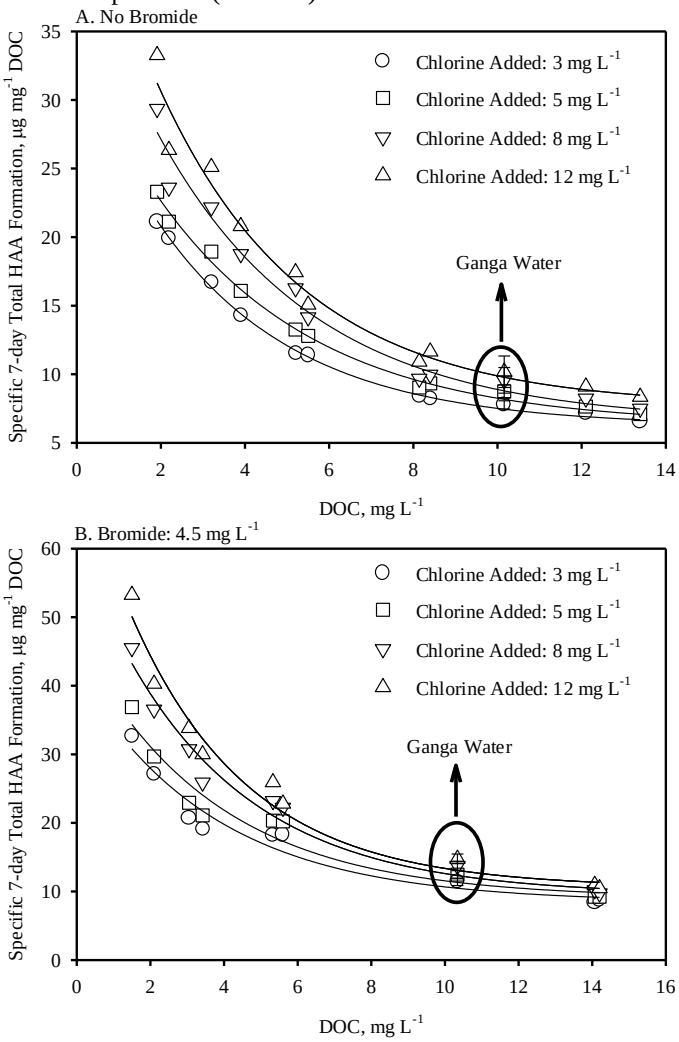

Fig 4.2:7-day Specific HAA formation of Diluted Wastewater and Ganga Riverwater at Various Applied Chlorine Doses.A) No Bromide; B) Bromide: 4.5 mg L^{-1}

Hence the specific HAA formation corresponding to the lowest DOC concentrations and the highest chlorine dose have beenultimate (i.e., 7-day) specific HAAs formation when the amount of chlorine added is in excess and hence not a limiting factor in HAAs formation.

In the case of HAAs with Bromide concentration (~5 mg/L) TCAA was found in maximum concentration with different concentrations of DOC followed by DCAA, BAA, CDBAA,CAA, BDCAA and BCAA (see Figure 4.3). Similar was with the case of Ganga river water. In the case of wastewater and Ganga river water without Bromide concentration TCAA was in maximum concentration followed by DCAA and CAA. Thus it was concluded that the presence of bromide in water samples favoured the formation of mixed and brominated HAAs.

The results presented in the previous section clearly show that the relative abundance of HAA components in a water sample is indifferent to the applied chlorine dose. This suggests that the formation potential of individual components of HAAs are proportional to the formation potential (FP) of the total HAA (THAA). Therefore, analysis of data in this section is focused on the formation potential of THAA.

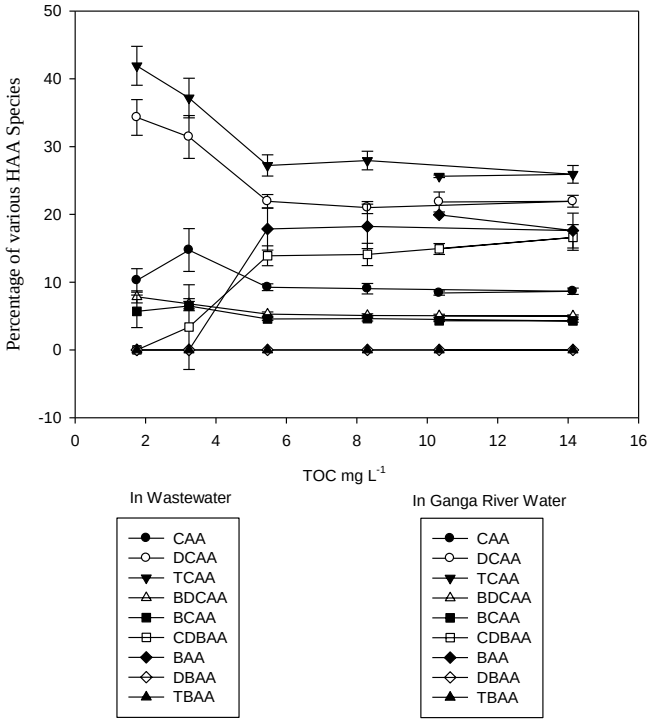

Fig 4.3: Percentage of various HAA species in different wastewaters and Ganga River water

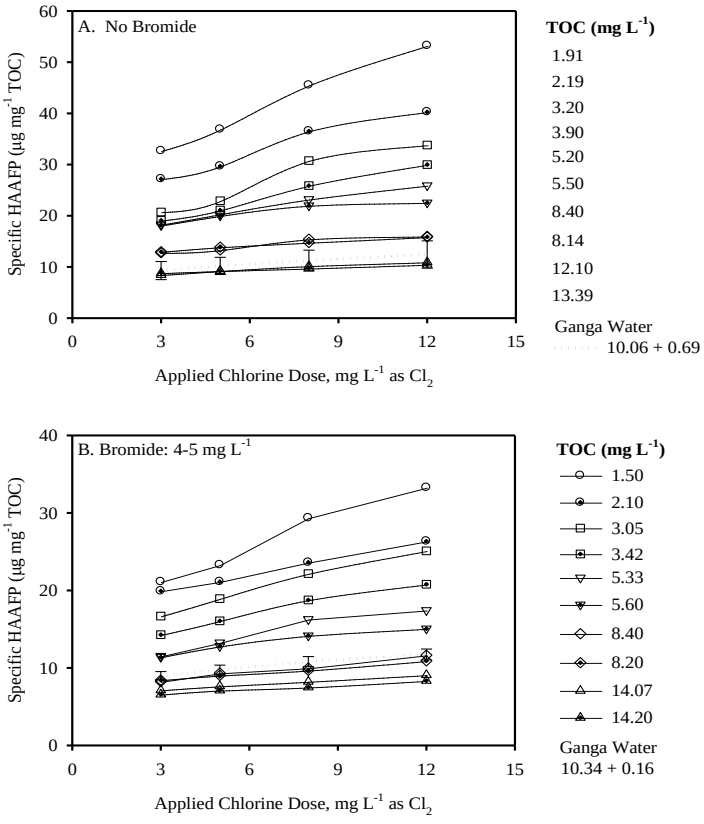

Fig. 4.4 Specific THAAFP as a function of Applied Chlorine Dose with No-Bromide and with Bromide Concentration

The THAAFP data obtained during this study was normalized by dividing with the TOC of the sample. These normalized values will henceforth be called specific THAAFP. Specific

THAAFP values without Bromide and with Bromide (4-5 mg/L) concentration (see **figure 4.4 and 4.5** respectively) for wastewater with different TOC and Ganga river water were plotted against the corresponding applied chlorine dose.
From the above figure it is noted that the Specific THAAFP for wastewater of different TOCs are of increasing in nature with increase in chlorine dose. Similar was the case with Ganga river water also.

Table 4.1 Alum dosing and corresponding turbidity in NTU

Alum Dose (mg/L)	Turbidity (NTU)-1	Turbidity - 2	Turbidity-3
0	18	17	16.1
20	10	11	9.6
40	7	8.3	6.3
60	7	7.4	6.8
80	6.8	7.6	6.9
100	7	7.2	7.1
120	9	9.5	9.8

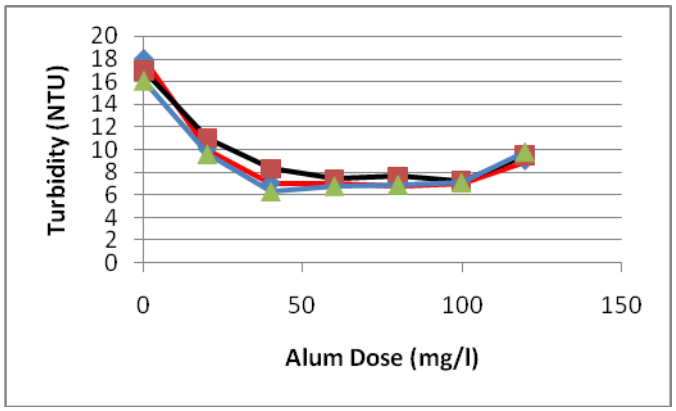

Fig. 4.5: Optimisation of Alum Dose

Different alum dosing was given in the raw Ganga river water and corresponding turbidity was measured with the help of turbidity meter. The optimum alum dose was noted after which there was no decrement in the turbidity. It was found as 60 mg/L as alum.

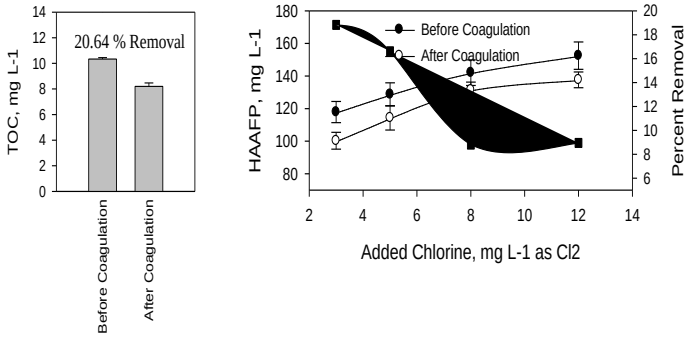

Fig. 4.6: Percentage removal of TOC, HAAFP of Raw Ganga river water with Bromide (4-5mg/L) concentration

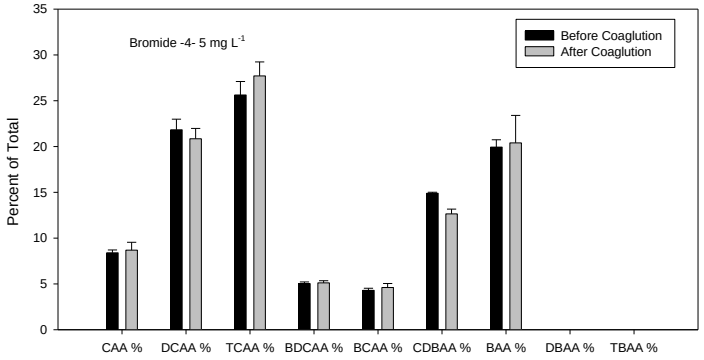

Fig. 4.7: Percentage of total HAA components before and after coagulation of Raw Ganga river water with Bromide (4-5mg/L) concentration

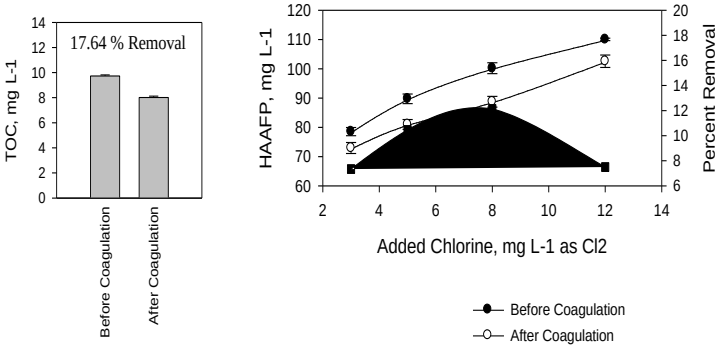

Fig. 4.8: Percentage removal of TOC, HAAFP of Raw Ganga river water without Bromide concentration

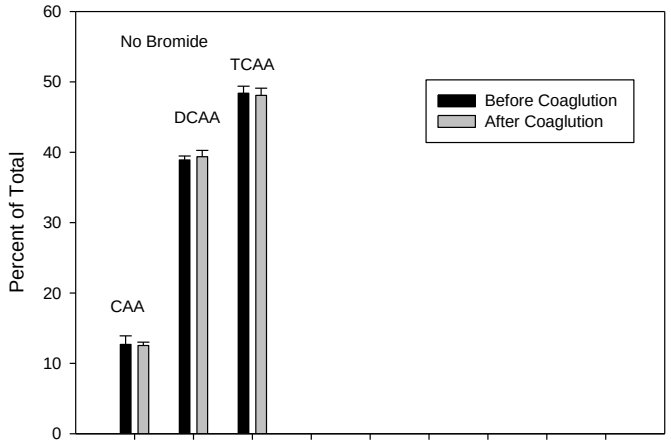

Fig. 4.9: Percentage of total HAA components before and after coagulation of Raw Ganga river water without Bromide concentration

From the above graph it was noted that percentage removal of HAAFP was varying 18-12 % in case of the presence of Bromide concentration and it was 5-12 % in the second case.

DISCUSSION

Nature of HAAs Precursors

DBP precursors, i.e., organic matter present in natural water supplies can be from, 1) allochthonous, i.e., of terrestrial origin (mainly humic substances), 2) autochthonous, i.e., derived from aquatic biological activity (mainly algal residues), or 3) derived from effluent discharges from anthropogenic sources (EffOM).

In water supplies unpolluted by anthropogenic activity, organic matter will primarily be of types 1 and 2, while in water supplies polluted by anthropogenic discharges, organic matter of types 2 and 3 will be predominant.

Sirivedhin and Gray [10] have shown clear differences in the structural characteristics of aquatic organic matter of different origins. Further, several studies have indicated that the extent of THM and HAA formation on chlorination of natural waters vary depending on the nature of DBP precursors, i.e., organic matter present in the water [10].

A compilation of specific THMFP and specific HAAFP values from various unpolluted surface water sources and also for humic and fulvic acids extracted from unpolluted surface waters is shown in Table 4.2.

Table 4.2 Literature Values of THAAFP from Surface Water Unpolluted by Anthropogenic Organic Carbon

	THAAFP (μg mg^{-1} DOC)
CROKE (Sirivedhin and Gray, 2005)[10]	Sample 1: 84.5 Sample 2: 118.1
SPSS (Sirivedhin and Gray, 2005) [10]	Sample 1: 55.4 Sample 2: 87.4
SPMG (Sirivedhin and Gray, 2005) [10]	Sample 1: 50.1 Sample 2: 73.6
Humic Acid (Reckhow et al., 1990) [11]	Sample 1: 170.0 Sample 2: 125.0
Fulvic Acid (Reckhow et al., 1990) [11]	Sample 1: 77.0 Sample 2: 75.0 Sample 1: 35.0 Sample 2: 30.0

A compilation of specific THAAFP values from various surface water sources polluted by organic carbon from anthropogenic sources (i.e., containing EffOM) is shown in **Table 5.2**.

Table 4.3 Literature Values of THMFP and HAAFP from Surface Waters Polluted by Anthropogenic Organic Carbon

	THAAFP (μg mg^{-1} DOC)
BCEFF (Sirivedhin and Gray, 2005) [10]	Sample 1: 20.9 Sample 2: 49.0
SPBC (Sirivedhin and Gray, 2005) [10]	Sample 1: 20.1 Sample 2: 50.7

Comparison of results presented in **Tables 4.2 and 4.3** clearly show that the specific THMFP and specific HAAFP was clearly lower in water containing EffOM.

As a part of this study, specific HAAFP was determined for diluted wastewater (DW) samples. Effluent organic matter (EffOM) was the primary source of DBP precursors in such samples. The specific THAAFP values were approximately 35 and 55 μg mg^{-1} DOC respectively in the presence and absence of bromide. As expected, these values are in rough agreement with the corresponding values in **Table 4.3** and much lower than the values presented in **Table 4.2**.

Based on the results of the study, it was also established that on chlorination, the HAA formation was roughly similar in Diluted waste water (DW) and Ganga water (GW) both in terms of the extent of HAAs formation and also in terms of the relative abundance of various HAA species (see **Figures 4.1 – 4.7)** formed. These results can be explained as follows. Analysis of Ganga River water quality upstream of Kanpur over the last decade clearly shows the river to be highly polluted with wastewater discharges from domestic and industrial effluents [12]. Further, studies have shown extensive evidence of algal activity in the Ganga River near Kanpur [13]. Considering the above facts, it is reasonable to assume that most organic matter present in the Ganga River in Kanpur is of anthropogenic ((EffOM) or autochthonous origin. The relative abundance of allochthonous organic matter, e.g., humic and fulvic acids in GW is consequently less as compared to organic matter from other sources.

Removal of HAA Precursors

Literature reports indicate that the efficiency of removal of DBP precursors by the coagulation process depend on the nature of the precursors. Musikavong et al. [14] reported that humic substances can be efficiently removed by coagulation. Cheng and Chi [15] observed that efficiency of the coagulation process vis-a-vis DOC removal declined after eutrophication. This suggests that organic matter of autochthonous origin may not be amenable to efficient removal through coagulation. Chow et al. observed that alum coagulation preferentially removed the higher molecular weight UV absorbing compounds whilst the remaining organic matter had lower apparent molecular weights (about 500-700 Daltons) and less UV absorbance. It was further observed those polysaccharides and their derivatives, which are mainly of autochthonous origin or are present as a result of anthropogenic pollution, were recalcitrant to removal with alum coagulation. These reports and other all suggest that while allochthonous organic matter, i.e., mainly humic substances are in general removed efficiently by the coagulation process, organic matter of autochthonous origin and EffOM may not be amenable to efficient removal by coagulation.

In the present study, coagulation experiments for HAA precursor removal from GW were conducted. DOC removals by coagulation were quite poor, viz., in the range of 15 – 20%. This is expected, since, as discussed before, the organic matter present in GW is probably of autochthonous origin and EffOM origin and hence not amenable to efficient removal by coagulation. As a consequence of the poor HAA precursor removal, THAAFP removal was also poor, i.e., generally below 15 percent in all cases.

5) CONCLUSIONS

Considering the nature of organic matter in GW in Kanpur as established during this study, it is reasonable to conclude that control of the DBPs concentration in this water cannot be achieved by optimizing DBPs precursor removals through the coagulation process during water treatment. The DBP precursors present in this water are not amenable to removal by coagulation. Consequently, other methods of DBPs precursor control, i.e., chlorination, biological filtration, etc. must be adopted for effective DBPs control in GW in Kanpur.

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